PATENT SPECIFICATION

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(54) METHOD AND APPARATUS FOR CONTINUOUSLY MONITORING TOTAL OXYGEN DEMAND

(71) We, PROCEDYNE CORPORATION, of 221 Somerset Street, New Brunswick, New Jersey 08903, United States of America; a corporation organised and existing under the laws of the State of New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to apparatus and method for analysing a continuously flowing sample stream of water or other fluid containing contaminants in order to determine the anticipated oxygen depletion of the water or other fluid due to oxidation of the contaminants in terms of the quantity of oxygen required to completely oxidize the same under high temperature reaction conditions, and to provide an instantaneous read-out of such content.

The determination of the total oxygen required to completely oxidize the contaminants present in an aqueous system offers a means of measuring the pollution level of the system. The basis for this measurement is that fact that the contaminants present in a lake, stream, river or any water supply gradually oxidize over a period of time causing the dissolved oxygen content of the water to drop. Since the oxygen thus consumed is replenished by natural means, such as by contact with the atmosphere, mechanical means, aeration ponds, and other agents, the rate of replenishment is limited by the particular situation involved. Since the oxidation of the contaminants is a major mechanism in the cleaning of the stream for re-use, the amount of oxygen required for oxidation of the contaminants is a measure of the difficulty of effecting a removal of the pollution. In addition, since fish and other aquatic life is strongly influenced by the oxygen level in the water, oxygen depletion due to higher rate of consumption in the oxidation reaction of contaminants relative to the rate of replenishment, has an important effect on this life and is clearly an important measure of the pollution level.

Since observation and/or analysis of the rate of natural oxidation of contaminants is not always a practical approach in determining the oxygen requirements of a polluted aqueous system, means have been sought to measure parameters which relate directly, or can be indirectly related to the anticipated oxygen depletion of the stream. One important parameter is the total oxygen demand (TOD) which has been defined as the oxygen required to completely oxidize the contaminants present in the stream. This is normally accomplished by subjecting the stream to high temperature oxidizing conditions so that the results may be obtained rapidly and conveniently.

Since the oxidizing conditions are not similar to those which are encountered when the stream is oxidized through its natural experience, TOD is not identically a measure of anticipated oxygen depletion; however, it has been shown to be relatable to the anticipated oxygen depletion of the system and is therefore regarded as an important measure of the degree of pollution. In addition, the particular means of inducing this rapid high temperature oxidation (i.e. the details of the oxidizing reactor with respect to reaction efficiency as well as the operating conditions) can influence the results obtained when determining TOD.

The present invention has for its principal object the provision of apparatus and a method for continuously analyzing the total oxygen demand (TOD) of contaminants contained in a fluid medium, by measuring the quantity of oxygen required to completely oxidize the contaminants by subjecting the stream to a specific high tempera-

ture reactor of high efficiency. The apparatus is automatic and functions continuously without requiring manual pre-treatment of a sample. Thus, there is obtained a parameter which can be related to the anticipated depletion of the oxygen content of bodies of water into which the waste streams are discharged. The parameter thus obtained can be termed total oxygen demand (TOD) as referred to in pollution evaluation: however, the particular values can be specific to the particular technique embodied in this invention.

According to the present invention there is provided apparatus for continuously determining the total quantity of oxidisable contaminants in an aqueous stream in terms of the total quantity of oxygen required to completely oxidize the contaminants present, comprising: means for providing a sample of the stream at a predetermined constant volumetric rate, a continuously-operating, fluidized-bed, bottom-fed reactor to receive and vaporize the sample means to heat the reactor, means to remove entrained solids from the reactor off-take, means to deliver oxygen to the reactor at a predetermined volumetric rate proportional to the volumetric rate of flow of the sample to oxidise the oxidisable contaminants in the sample, means to remove water from said off-take and means for determining the concentration of oxygen in the off-gas from the reactor.

Further according to the present invention there is provided a process for continuously determining the total quantity of oxidisable contaminants in an aqueous stream which comprises the steps of:

(a) continuously delivering a sample of the stream to a fluidized-bed, continuously-operating, bottom-fed reactor at a constant predetermined volumetric rate;

(b) continuously delivering oxygen to the reactor at a predetermined volumetric rate having a predetermined proportion to the volumetric rate of flow of the sample, said oxygen rate being at least sufficient to oxidize all of the carbon;

(c) heating the reactor to provide an operating temperature;

(d) passing the reactor off-gas through a separator to remove entrained solids;

(e) passing the reactor off-gas through a condenser to eliminate remaining water;

(f) analyzing the resulting gas mixture to determine the oxygen uptake of the system by determination of the difference between the quantity of oxygen delivered to the reactor and residual oxygen in the off-gas.

Also according to the present invention there is provided a process for continuously determining the total quantity of oxidisable contaminants in an aqueous stream which comprises:

(a) continuously extracting a sample from the stream;

(b) causing the sample to flow to a continuously-operating, fluidized-bed, bottom-fed reactor at a predetermined volumetric rate;

(c) injecting oxygen into the sample at a predetermined volumetric rate at least sufficient to oxidise all of the carbon;

(d) heating the oxygenated sample in the reactor to oxidize the carbon;

(e) scrubbing entrained solids and water from the reactor off-gas;

(f) determining the difference between the quantity of oxygen added to the sample stream and the residual quantity thereof in the off-gas;

(g) converting said difference to equivalent carbon as a measure of the contaminants in the aqueous stream.

The present invention concerns determination of the total oxygen demand (TOD) in a sample stream without manual pre-treatment or manipulation, by measuring the quantity of oxygen required to completely oxidize the same in a specific high temperature, high efficiency, oxidizing reactor. The apparatus and method, sometimes referred to for brevity as a TOD monitor, i.e. total oxygen demand monitor, comprises the following:

1. Metering the waste stream at a known predetermined rate into a fluidized solids reactor typically operating above 1500°F. Metering is typically accomplished using a liquid metering pump.
2. Concurrently, oxygen is metered to the reactor at a known predetermined rate typically controlled by a precision metering valve and measured by a precision rotometer, or precision pressure gage and flow controller, e.g. an orifice or other commercial device.
3. The incinerator reactor is a fluidized-solids reactor typically heated by electric heaters, together with temperature-regulating means. The fluidization gas is the mixture of vaporized waste stream, combustion products and unreacted oxygen.
4. The reactor off-gas is passed through a cyclone separator for removal of entrained solids, a condenser for removal of most of the water, and other devices for removal of compounds which are vaporizable at the combustion temperature and which could interfere with the oxygen measurement. The resulting gas mixture is continuously fed to an oxygen analyzer where the oxygen concentration in the off-gas is determined.
5. The difference in oxygen accounted for in the off-gas and the oxygen fed into the reactor is the oxygen uptake of the system (TOD).

ture reactor of high efficiency. The apparatus is automatic and functions continuously without requiring manual pre-treatment of a sample. Thus, there is obtained a parameter which can be related to the anticipated depletion of the oxygen content of bodies of water into which the waste streams are discharged. The parameter thus obtained can be termed total oxygen demand (TOD) as referred to in pollution evaluation; however, the particular values can be specific to the particular technique embodied in this invention.

According to the present invention there is provided apparatus for continuously determining the total quantity of oxidisable contaminants in an aqueous stream in terms of the total quantity of oxygen required to completely oxidize the contaminants present, comprising: means for providing a sample of the stream at a predetermined constant volumetric rate, a continuously-operating, fluidized-bed, bottom-fed reactor to receive and vaporize the sample means to heat the reactor, means to remove entrained solids from the reactor off-take, means to deliver oxygen to the reactor at a predetermined volumetric rate proportional to the volumetric rate of flow of the sample to oxidise the oxidisable contaminants in the sample, means to remove water from said off-take and means for determining the concentration of oxygen in the off-gas from the reactor.

Further according to the present invention there is provided a process for continuously determining the total quantity of oxidisable contaminants in an aqueous stream which comprises the steps of:

(a) continuously delivering a sample of the stream to a fluidized-bed, continuously-operating, bottom-fed reactor at a constant predetermined volumetric rate;

(b) continuously delivering oxygen to the reactor at a predetermined volumetric rate having a predetermined proportion to the volumetric rate of flow of the sample, said oxygen rate being at least sufficient to oxidize all of the carbon;

(c) heating the reactor to provide an operating temperature;

(d) passing the reactor off-gas through a separator to remove entrained solids;

(e) passing the reactor off-gas through a condenser to eliminate remaining water;

(f) analyzing the resulting gas mixture to determine the oxygen uptake of the system by determination of the difference between the quantity of oxygen delivered to the reactor and residual oxygen in the off-gas.

Also according to the present invention there is provided a process for continuously determining the total quantity of oxidisable contaminants in an aqueous stream which comprises:

(a) continuously extracting a sample from the stream;

(b) causing the sample to flow to a continuously-operating, fluidized-bed, bottom-fed reactor at a predetermined volumetric rate;

(c) injecting oxygen into the sample at a predetermined volumetric rate at least sufficient to oxidise all of the carbon;

(d) heating the oxygenated sample in the reactor to oxidize the carbon;

(e) scrubbing entrained solids and water from the reactor off-gas;

(f) determining the difference between the quantity of oxygen added to the sample stream and the residual quantity thereof in the off-gas;

(g) converting said difference to equivalent carbon as a measure of the contaminants in the aqueous stream.

The present invention concerns determination of the total oxygen demand (TOD) in a sample stream without manual pretreatment or manipulation, by measuring the quantity of oxygen required to completely oxidize the same in a specific high temperature, high efficiency, oxidizing reactor. The apparatus and method, sometimes referred to for brevity as a TOD monitor, i.e. total oxygen demand monitor, comprises the following:

1. Metering the waste stream at a known predetermined rate into a fluidized solids reactor typically operating above 1500°F. Metering is typically accomplished using a liquid metering pump.
2. Concurrently, oxygen is metered to the reactor at a known predetermined rate typically controlled by a precision metering valve and measured by a precision rotometer, or precision pressure gage and flow controller, e.g. an orifice or other commercial device.
3. The incinerator reactor is a fluidized-solids reactor typically heated by electric heaters, together with temperature-regulating means. The fluidization gas is the mixture of vaporized waste stream, combustion products and unreacted oxygen.
4. The reactor off-gas is passed through a cyclone separator for removal of entrained solids, a condenser for removal of most of the water, and other devices for removal of compounds which are vaporizable at the combustion temperature and which could interfere with the oxygen measurement. The resulting gas mixture is continuously fed to an oxygen analyzer where the oxygen concentration in the off-gas is determined.
5. The difference in oxygen accounted for in the off-gas and the oxygen fed into the reactor is the oxygen uptake of the system (TOD).

A sample of the waste is diverted from the main stream for flow through a conduit 10, the flow rate of the sample being predetermined by means of a metering pump 12. The stream continues through a conduit 13, back-pressure and check valve 14 and conduit 16 to the bottom of a fluidized solids reactor 18. The valve 14 is designed to minimize the effect of pressure fluctuations in the reactor on the precision of the liquid feed rate.

Prior to delivery of the waste stream to the reactor, it is joined by a stream of oxygen flowing through conduits 22, 24 and 26; there being a precision flow gauge 25 interposed to measure the predetermined rate of flow of the oxygen. The metering pump is typically an electrically-driven, positive-displacement pump designed to deliver the sample at a highly accurate, constant rate. In the example, the flow gage is typically a rotameter Model 10A1367A13A, as supplied by Fischer & Porter, located at Wor-minster, Pennsylvania. A check valve 27 prevents back flow of the waste stream into the line 24.

The reactor 18 is of the fluidized-bed type and comprises a bottom feed pipe through which the water-oxygen mixture is admitted to the bed 18a, e.g. comprising particles of aluminum oxide or other material which is incombustible at the operating temperature of the bed, e.g. 1500°F or higher. Heat is supplied to the bed by means of sheathed electrical resistance heaters 25b. The temperature of the bed is regulated in any well-known manner, e.g. by the use of a thermocouple 29 and controller 31.. In addition, the reactor is equipped with a high temperature alarm which is arranged to interrup current to the heaters if the temperature exceeds a pre-set high. All electrical connections are indicated diagrammatically. The fluidizing gas fed to the bed comprises a mixture of vaporized waste stream, combustion products and unreacted oxygen..

Off-gas from the reactor is delivered to a condenser 41 via a line 42, for removal of most of the water and thence through a pipe 44, to a cyclone separator 45 for removal of entrained solids. Such liquid and solids are received in a vessel 51 wherefrom they are disposed of to waste at 52, the liquid retained in the vessel serving as a water seal against loss of reactor gas. The resulting mixture of gases leaving the cyclone separator at 54 is additionally scrubbed in a condenser 55 and then passed, through line 56, to a CO_2 scrubber and dryer 57 to an oxygen analyzer 58, where the concentration of oxygen in the off-gas is determined.

Since the initial delivery of oxygen is determined at a known rate by means of the flow gauge 25 and the analyzer 58 provides information on residual oxygen, the difference is the quantity of oxygen taken up in the system, i.e. total oxygen demand

In a practical embodiment of the invention, the following operating criteria were employed:

Feed of waste stream:	5 ml/min.
Oxygen feed:	40 ml/min.
Reactor temperature:	1630°F
Solids in reactor:	150 mesh, Al_2O_3

WHAT WE CLAIM IS:—

1. Apparatus for continuously determining the total quantity of oxidisable contaminants in an aqueous stream in terms of the total quantity of oxygen required to completely oxidize the contaminants present, comprising: means for providing a sample of the stream at a predetermined constant volumetric rate, a continuously-operating, fluidized-bed, bottom-fed reactor to receive and vaporize the sample, means to heat the reactor, means to remove entrained solids from the reactor off-take, means to deliver oxygen to the reactor at a predetermined volumetric rate proportional to the volumetric rate of flow of the sample to oxidise the oxidisable contaminants in the sample, means to remove water from said off-take and means for determining the concentration of oxygen in the off-gas from the reactor.

2. Apparatus for continuously determining the total quantity of oxidisable contaminants in an aqueous stream, substantially as hereinbefore described with reference to and as illustrated in the accompanying drawing.

3. A process for continuously determining the total quantity of oxidisable contaminants in an aqueous stream which comprises the steps of:-

(a) continuously delivering a sample of the stream to a fluidized-bed, continuously-operating, bottom-fed reactor at a constant predetermined volumetric rate;

(b) contiuously delivering oxygen to the reactor at a predetermined volumetric rate having a predetermined proportion to the volumetric rate of flow of the sample, said oxygen being at least sufficient to oxidize all of the carbon;

(c) heating the reactor to provide an operating temperature;

(d) passing the reactor off-gas through a separator to remove entrained solids;

(e) passing the reactor off-gas through a condenser to eliminate remaining water;

(f) analyzing the resulting gas mixture to determine the oxygen uptake of the system by determination of the difference between the quantity of oxygen delivered to the reactor and residual oxygen in the off-gas

4. A process as claimed in claim 3, in

which the oxygen is continuously combined with the sample.

5. A process for continuously determining the total quantity of oxidisable contaminants in an aqueous stream which comprises:

(a) continuously extracting a sample from the stream;

(b) causing the sample to flow to a continuously-operating fluidized-bed, bottom-fed reactor at a predetermined volumetric rate;

(c) injecting oxygen into the sample at a predetermined volumetric rate at least sufficient to oxidise all of the carbon;

(d) heating the oxygenated sample in the reactor to oxidize the carbon;

(e) scrubbing entrained solids and water from the reactor off-gas;

(f) determining the difference between the quantity of oxygen added to the sample stream and the residual quantity thereof in the off-gas;

(g) converting said difference to equivalent carbon as a measure of the contaminants in the aqueous stream.

6. A process for continuously determining the total quantity of oxidisable contaminants in an aqueous stream as claimed in any one of claims 3 to 5, substantially as hereinbefore described.

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1392405 COMPLETE SPECIFICATION

1 SHEET This drawing is a reproduction of the Original on a reduced scale